भारतीय मानक
Indian Standard

IS 2744 : 2022

1-नैफ्थिलअमीन — विशिष्टि

(*दूसरा पुनरीक्षण*)

1-Napthylamine — Specification

(*Second Revision*)

ICS 71.080.30

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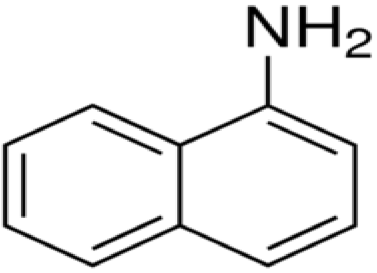
July 2022 **Price Group 8**

Dye Intermediates Sectional Committee, PCD 26

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

1-Napthylamine is an important material in the dyestuff industry. It is also used as an intermediate in organic synthesis. The material is represented by the following structural formula:



1-NAPTHYLAMINE
[MOLECULAR MASS= 143.2]
[CAS REGISTRY NO.:134-32-7]

This standard was first published in 1964 and subsequently revised in 1984. However, the research and developmental work in late 70s have shown that the isomer 2-naphthylamine is carcinogenic in nature, that is cancer causing and is invariably present with the material, namely, 1-naphthylamine. Therefore, in 1984, the Committee decided to revise it first time with a view to restrict the maximum permissible limit to a much lower level. The consequential changes in the other requirements of the material have also been suitably given effect.

In this (second) revision, requirement value of total amine content (on molecular mass 143.2 basis), percent by mass, *Min* has been updated. Along with that additional requirement of chlorobenzenes and chlorotoluenes have also been incorporated considering the environmental hazard of these derivatives. Considering these facts and development in latest analytical techniques, Committee incorporated high performance liquid chromatography method for the determination of 1-naphthylamine content and impurities of chlorobenzene's (CB's) and chlorotoluene's (CT's) by gas chromatography mass spectrometer. Amendment 1 had also been incorporated.

The composition of the Committee responsible for formulation of this standard is given in Annex G.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

SPECIFICATION FOR 1-NAPHTHYLAMINE

(Second Revision)

1 SCOPE

This standard prescribes the requirements, methods of sampling and testing for 1-naphthylamine.

2 REFERENCES

The following standards contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards given below:

IS No	*Title*
1070 : 1992	Reagent grade water — Specification (*third revision*)
2480 (Part 1) : 1983	Specification for general purpose glass thermometers: Part 1 Solid stem thermometers (*second revision*)
2552 : 1989	Steel drums (galvanized and ungalvanized) — Specification (*third revision*)
5299 : 2001	Methods of sampling and tests for dye intermediates (*first revision*)

3 REQUIREMENTS

3.1 Description

The material shall be in the form of colourless or light brown flakes or lumps.

3.2 The material shall also comply with the requirements given in Table 1, when tested according to the methods prescribed in col 4 and col 5 of Table 1.

Table 1 Requirements for 1-Naphthylamine

(*Clauses* 3.2, 5.3.1, 5.3.2 *and* 6.2)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex	Clause No. in IS 5299
(1)	(2)	(3)	(4)	(5)
i)	Crystallizing point, °C, *Min*	48	A	—
ii)	Matter insoluble in dilute hydrochloric acid, percent by mass, *Max*	0.2	—	11.1
iii)	Total amine content (on molecular mass 143.2 basis), percent by mass, *Min*	98.0	B	—
iv)	Assay (by HPLC), percent by mass, *Min*	98.0	C	—
v)	2-naphthylamine content, percent by mass, *Max*	0.5	D	—
vi)	Sum of chlorobenzene’s and chlorotoluene’s, ppm, *Max*	50.00	E	—

4 PACKING AND MARKING

4.1 Packing

The material shall be suitably packed either in wooden barrels, multiwalled paper sacks or suitable drums (*conforming to* IS 2552).

4.2 Marking

4.2.1 The containers shall be securely closed and shall bear legibly and indelibly the following information:

a) Name of the material;
b) Name of manufacturer and his recognized trade-mark, if any;
c) Net mass of material;
d) Month and year of the manufacture;
e) Batch or lot number; and
f) Any other statutory requirements.

4.2.2 Each container shall bear the minimum cautionary notice worded as under:

WARNING
DUST AND VAPOUR HARMFUL.
REPEATED
ABSORPTION MAY RESULT IN
BLADDER TUMORS

4.2.3 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed there under, and the products may be marked with the Standard Mark.

5 SAMPLING

5.1 Preparation of Test Samples

The method of drawing representative samples of the material shall be as prescribed in Annex F.

5.2 Number of Tests

5.2.1 Tests for the determination of crystallizing point, total amine content and

2-naphthylamine content shall be conducted on the individual samples separately.

5.2.2 Tests for the determination of the remaining characteristic, that is, matter insoluble in dilute hydrochloric acid, assay and sum of chlorobenzene’s and chlorotoluene’s shall be conducted on the composite sample.

5.3 Criteria for Conformity

5.3.1 *For Individual Samples*

The lot shall be declared as conforming to the requirements of crystallizing point, total amine content and 2-naphthylamine content if each of the individual test results satisfies the relevant requirement given in Table 1.

5.3.2 *For Composite Samples*

For declaring the conformity of the lot to the requirements of all other characteristics (**5.2.2**), the the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

6 TESTS

6.1 Preparation of Sample

Mix the sample well by rotating the bottle several times. Transfer a portion immediately into a wide mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acid or alkaline fumes. Use this prepared sample for tests.

6.2 Tests shall be carried out on the prepared sample (**6.1**) according to the methods prescribed in col 4 and col 5 of Table 1.

6.3 Quality of Reagents

Unless specified otherwise, ‘pure chemicals’ and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals’ shall mean chemicals that do not contain impurities which affect the results of analysis.

ANNEX A

[*Table* 1, *Sl No.* (i)]

DETERMINATION OF CRYSTALLIZING POINT

A-1 APPARATUS

A-1.1 The apparatus of the shape, dimensions and tolerances given in Fig. 1, consists of the following.

A-1.1.1 Outer Glass Test Tube

Serves as an air jacket and is weighted with lead shots or a similar loading material. It is provided with a cork through which the inner tube (*see* **A-1.1.2**) is held in position.

A-1.1.2 Inner Glass Test Tube

Fitted with a cork which carries a stirrer in the form of a loop of glass with a glass stem and the thermometer placed in the center within the tube and the glass loop. The bottom of the bulb of the thermometer shall be about 10 mm from the bottom of the inner tube. The cork is so fixed that the immersion mark on a thermometer is in level with the top of the cork.

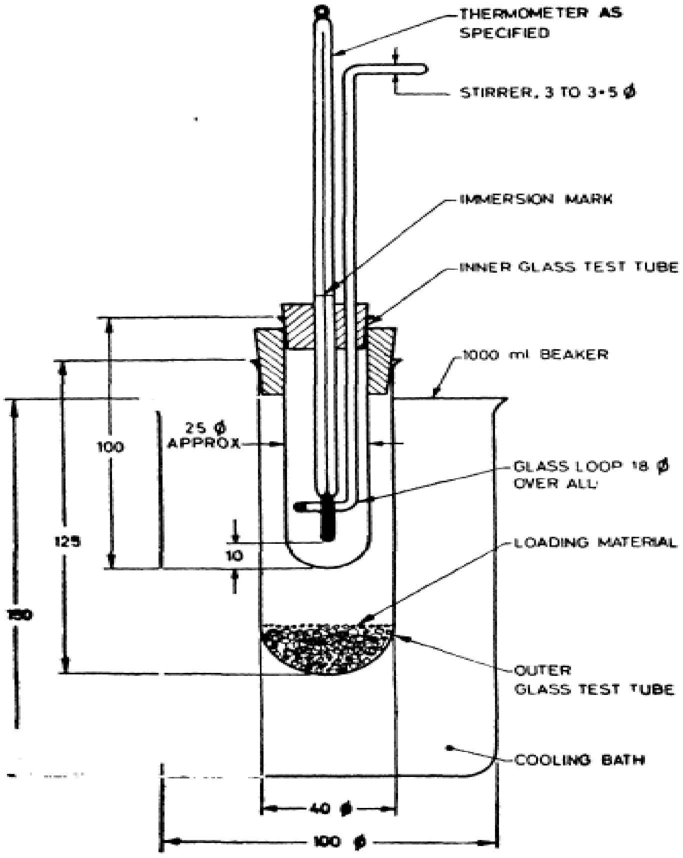


FIG 1. APPARATUS FOR THE DETERMINATION OF CRYSTALLIZATION POINT

A-1.1.3 Cooling Bath, 1 000 m1 beaker about 150 mm in height. The level of the cooling liquid in the bath shall be at least as high as the level of the sample in the inner tube.

A-1.1.4 Thermometer [*see* IS 2480 (Part 1)]

A-2 PROCEDURE

A-2.1 Remove the inner tube of the apparatus from its jacket and introduce about 20 g of the material. Immerse the tube partially in a bath having a temperature of about 5 °C above the crystallizing point, until all but the last traces of crystal have melted. Replace the inner tube in its jacket and assemble the apparatus as shown in Fig. 1, with the cooling bath between 6 °C and 8 °C below the expected crystallizing point.

A-2.2 Take the thermometer readings at intervals of half a minute, with continuous and gentle stirring. This operation being so conducted that seed crystal is present as the temperature of the sample falls to that at which crystallization commences. The highest temperature reached during solidification is regarded as the crystallizing point. Usual thermometer corrections shall be made.

A-2.3 If super cooling should take place, the constant temperature should be observed immediately after the temperature-rise. A temperature-rise of 1 °C shall be regarded as the maximum allowable.

A-2.4 If a constant temperature is not obtained over the first five readings after the rise in temperature, six readings shall be taken commencing with the point at which the maximum temperature is first attained. The readings shall be plotted on graph paper against time intervals and a straight line drawn to lie evenly between the first and second and between the fifth and sixth of the six points just mentioned. This line shall be produced backwards until it intersects the portion of the curve before the temperature-rise. The point of intersection shall in this case be taken as the crystallizing point.

ANNEX B

[*Table* 1, *Sl No.* (iii)]

TOTAL AMINES CONTENT BY DIAZOTIZATION METHOD

B-1 GENERAL

1-Naphthylamine is estimated by direct titration with standard sodium nitrite solution, using starch potassium iodide paper as indicator.

B-2 REAGENTS

B-2.1 Sodium Nitrite Solution, approximately 0.2 N freshly standardized.

B-2.2 Starch and Potassium Iodide Papers

B-3 PROCEDURE

B-3.1 Heat 750 ml water to 80 °C. Add 81.5 ml concentrated hydrochloric acid. Mix and then add 14.32 g of the material. Stir until dissolved. The solution shall be clear with no globules of oil. The solution is used for the assay of 1-naphthylamine.

B-3.2 Solution obtained under **B-3.1** above shall be made up to 1 000 ml and 100 ml aliquot shall be taken and cooled with crushed ice to 50 °C. While stirring mechanically, titrate with sodium nitrite solution. Titrate as rapidly as possible to distinct faint blue ring test on a starch and potassium iodide paper. The final test shall be obtained repeatedly during a period of 10 min without further addition of nitrite.

B-4 CALCULATION

$$\text{Total Amines content percent} = \frac{143.2 \text{ x } V \text{ x } N}{M} \text{ x } 100$$

where

V = volume, of sodium nitrite solution, ml;

N = normality of sodium nitrite; and

M = mass of material taken for the test, g.

ANNEX C

[*Table* 1, *Sl No.* (iv)]

DETERMINATION OF 1-NAPHTHYLAMINE (α-NAPTHYLAMINE) (ASSAY) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

C-1 GENERAL

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture

C-2 APPARATUS

C-2.1 Binary Gradient Liquid Chromatography System, capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

C-2.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

C-3 REAGENT

C-3.1 Acetonitrile, HPLC grade.

C-3.2 Water, HPLC grade.

C-3.3 Tetra-butylammonium Hydrogen Sulphate

C-3.4 α**- Naphthylamine Standard**

C-4 STANDARD PREPARATION

Weigh accurately 0.050 0 g standards α-naphthylamine in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

C-5 SAMPLE PREPARATION

Weigh accurately 0.050 0 g sample in 100 ml volumetric flask. Dissolve in acetonitrile and make it up to the mark with acetonitrile.

C-6 BUFFER PREPARATION

0.2 percent tetra-butyl ammonium hydrogen sulphate in HPLC grade Water.

C-7 FLOW RATE, 1.00 ml/min.

C-8 MOBILE PHASE

Acetonitrile	Buffer
35	65

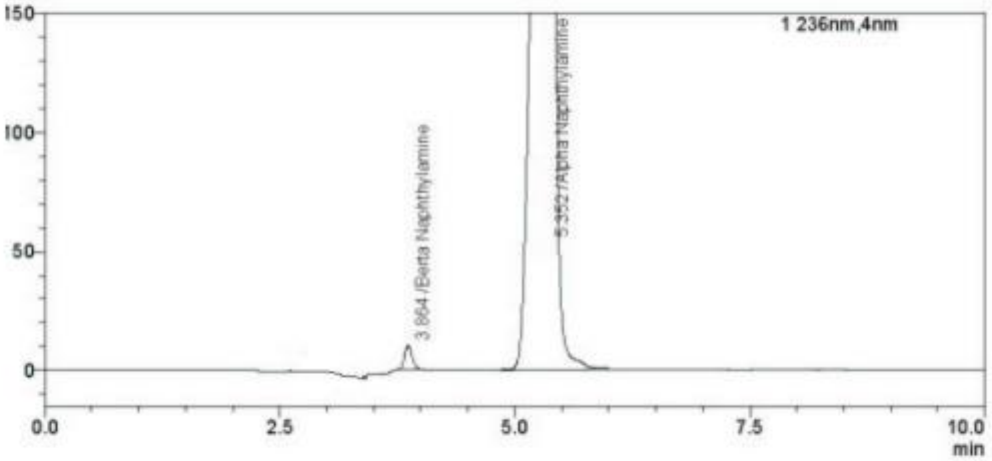
C-9 INJECTION VOLUME, 2 µl.

C-10 COLUMN OVEN TEMPERATURE, 40 ˚C.

C-11 WAVELENGTH, 236 nm.

C-12 PEAK TIME

α-Naphthylamine: 4.10 min



C-13 CALCULATION

Calculate the peak area of individual constituent pertaining to α-naphthylamine on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure α-naphthylamine.

$$\alpha - \text{Naphthylamine, percent} = \frac{(A_2 \times V_1 \times W_1 \times B_2)}{(A_1 \times V_2 \times W_2 \times B_1)} \text{ x } 100$$

where

A_1 = area of standard α-naphthylamine;

V_1 = injection volume of standard α-naphthylamine;

W_1 = weight of standard α-naphthylamine;

B_1 = total volume of standard α-naphthylamine;

A_2 = area of α-naphthylamine peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

ANNEX D

[*Table* 1, *Sl No.* (v)]

DETERMINATION OF 2-NAPHTHYLAMINE (*β*-NAPTHYLAMINE) CONTENT IN 1-NAPHTHYLAMINE (*α*-NAPTHYLAMINE) BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY

D-1 GENERAL

High-performance liquid chromatography or high-pressure liquid chromatography (HPLC) is a chromatographic method that is used to separate a mixture of compounds in analytical chemistry and biochemistry so as to identify, quantify or purify the individual components of the mixture.

D-2 APPARATUS

D-2.1 Binary Gradient Liquid Chromatography System, capable of being operated under conditions suitable for resolving the individual constituents into distinct peak may be used.

D-2.2 Column

C18 column of 100 Å with length 250 m, internal diameter 4.6 mm and particle size 5 µm or equivalent.

D-3 REAGENT

D-3.1 Acetonitrile, HPLC grade.

D-3.2 Water, HPLC grade.

D-3.3 Tetra-butyl Ammonium Hydrogen Sulphate

D-3.4 *β*-Naphthylamine Standard

D-4 STANDARD PREPARATION

Solution A — Weigh accurately 0.050 0 g standard *β*-naphthylamine in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetoniltrile.

Take 1 ml of solution A in 10 ml volumetric flask and dilute it up to the mark with acetonitrile.

D-5 SAMPLE PREPARATION

Weigh accurately 0.500 0 g sample in 100 ml volumetric flask. Dissolve it in acetonitrile and make it up to the mark with acetonitrile.

D-6 BUFFER PREPARATION

0.2 percent tetra-butylammonium hydrogen sulphate in HPLC grade water.

D-7 FLOW RATE, 1.00 ml/min.

D-8 MOBILE PHASE

Acetonitrile	Buffer
35	65

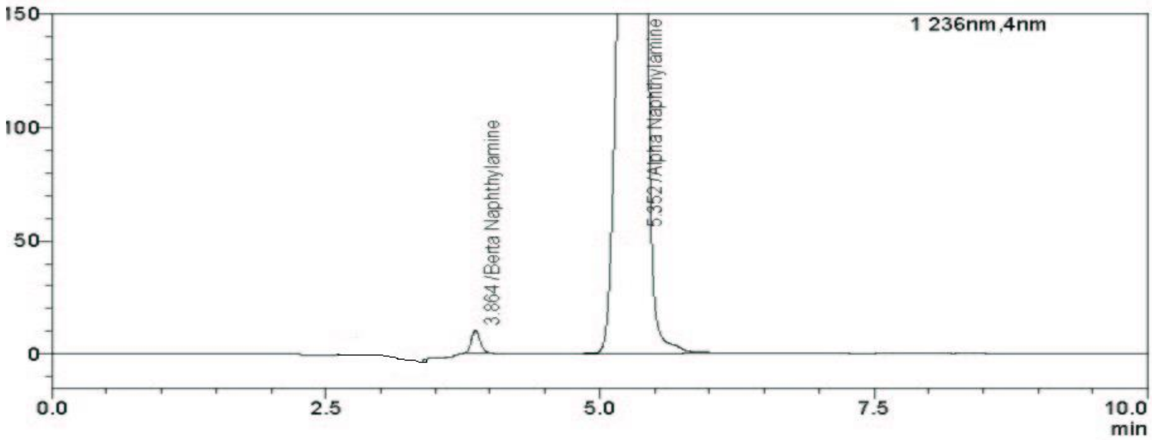
D-9 INJECTION VOLUME, 2 µl.

D-10 COLUMN OVEN TEMPERATURE, 40 °C.

D-11 WAVELENGTH, 236 nm.

D-12 PEAK TIME

α- Naphthylamine:	5.35 min
β-Naphthylamine:	3.86 min



D-13 CALCULATION

Calculate the peak area of individual constituent pertaining to β-naphthylamine on the chromatogram of the material. The concentration of the constituent may be obtained on the basis of peak area on chromatogram obtained with known amount of pure β-naphthylamine.

$$\beta - \text{naphthylamine, percent} = \frac{(A_2 \times V_1 \times W_1 \times B_2)}{(A_1 \times V_2 \times W_2 \times B_1)} \text{ x } 100$$

where

A_1 = area of standard β-naphthylamine;

V_1 = injection volume of standard β-naphthylamine;

W_1 = weight of standard β-naphthylamine;

B_1 = total volume of standard β-naphthylamine;

A_2 = area of β-naphthylamine peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

ANNEX E

[*Table* 1, *Sl No.* (vi)]

DETERMINATION OF CHLOROBENZENE’S AND CHLOROTOLUENE’S CONTENT BY GAS CHROMATOGRAPHY MASS SPECTROSCOPY

E-1 APPARATUS

Any gas chromatograph equipped with a mass spectroscopy (MS) detector, a split/splitless injector and a suitable electronic integrator/software can be used can be used with following accessories and operating condition:

E-1.1 Column, 65 percent dimethylpolysiloxane and 35 percent phenyl phase with length 30 m, internal diameter 0.25 mm, film thickness 0.25 µm and column maximum temperature 340 ^{0}C or equivalent

E-2 INJECTION PORT INFORMATION

Injection Mode	*Temperature*	*Column Flow*	*Purge Flow*	*Split Ratio*	*Injection Volume*	*Carrier Gas*
Split	240 °C	1.00 ml/min	3.0 ml/min	2.0	1 µl	Helium

E-3 COLUMN OVEN INFORMATION

Initial Temperature	*Equilibration time*	*Total Program Time*
40 °C	3.00	36 min

E-4 COLUMN OVEN TEMPERATURE PROGRAM

Heating Rate (°C /min)	*Temperature(°C)*	*Hold Time (min)*
—	40	0.5
5	140	1
20	290	6

E-5 DETECTOR INFORMATION

Detector	*Iron Source Temperature*	*Interface Temperature*	*Solvent Cut Time*	*Detector Voltage*	*Threshold*
MS	230 °C	280 °C	3 min	Relative + 0.3 kV	100

Start Time	*End Time*	*Acquire mode*	*M/Z*
3	11	SIM	63,77,91,112,114,126
11	15	SIM	65,91,111,114,146,148
15	20	SIM	125,127,145,160,180,182
20	26.5	SIM	159,161,194,196,214,218,240,250,252
26.5	29.5	SIM	229,284,286

E-6 AUTO INJECTION INFORMATION

Rinses with Solvent (pre Run)	*Rinses with Solvent (post Run)*	*Rinses with Sample*	*Plunger speed*	*Viscosity Comp. Temperature*	*Plunger Speed*	*Syringe inserting Speed*
0	5	2	High	0.2 s	High	High

NOTE — The above gas chromatographic (GC) conditions are suggestive. However, any GC method having difference in detector, column packing material and type (like packed/capillary, diameter, length, film thickness etc), calibration technique (internal standard, external standard, area normalization, percent area etc), carrier gas (He, H_2, N_2) may be used with applicable GC operating parameters, provided standardization and calibration of the components is established after setting GC parameters for the resolution and accuracy level as specified in this standard.

E-7 REAGENT

E-7.1 Monochlorobenzene [MCB]

E-7.2 1,2-dichlorobenzene [ODCB]

E-7.3 1,3-dichlorobenzene [MDCB]

E-7.4 1,4-dichlorobenzene [PDCB]

E-7.5 1,2,3-trichlorobenzene [1,2,3-TCB]

E-7.6 1,2,4-trichlorobenzene [1,2,4-TCB]

E-7.7 1,3,5-trichlorobenzene [1,3,5-TCB]

E-7.8 1,2,3,4-tetrachlorobenzene [1,2,3,4-TeCB]

E-7.9 1,2,3,5-tetrachlorobenzene [1,2,3,5-TeCB]

E-7.10 1,2,4,5-tetrachlorobenzene [1,2,4,5-TeCB]

E-7.11 Pentachlorobenzene [PCB]

E-7.12 Hexachlorobenzene [HCB]

E-7.13 Toluene

E-7.14 2-chlorotoluene [2-CT]

E-7.15 3-chlorotoluene [3-CT]

E-7.16 4-chlorotoluene [4-CT]

E-7.17 2,3-dichlorotoluene [2,3-DCT]

E-7.18 2,4-dichlorotoluene [2,4-DCT]

E-7.19 2,5-dichlorotoluene [2,5-DCT]

E-7.20 2,6-dichlorotoluene [2,6-DCT]

E-7.21 3,4-dichlorotoluene [3,4-DCT]

E-7.22 2,3,6-trichlorotoluene [2,3,6-TCT]

E-7.23 2,4,5-trichlorotoluene [2,4,5-TCT]

E-7.24 Pentachlorotoluene [PCT]

E-7.25 Dichloromethane [DCM]

E-7.26 Methanol

E-8 RETENTION TIME OF CHLOROBENZENE’S AND CHLOROTOLUENE’S

Product Name	*Retention Time*	*M/Z*
MCB	6.59	112
MDCB	11.33	146
PDCB	11.68	146
ODCB	12.57	146
1,3,5 TCB	14.97	180
1,2,4 TCB	16.83	180
1,2,3 TCB	18.21	180
1,2,3,5 TeCB	21.04	216
1,2,4,5 TeCB	21.13	216
1,2,3,4 TeCB	22.88	216
Penta CB	24.93	250
Hexa CB	26.75	284

Product Name	*Retention Time*	*M/Z*
Toluene	6.59	112
2-CT	9.52	91
3-CT	9.59	91
4-CT	9.65	91
AlphaCT	12.36	91
2,4-DCT	14.67	125
2,6-DCT	14.96	125
2,5-DCT	14.91	125
3,4-DCT	15.87	125
2,3-DCT	15.97	125
2,4,5-TCT	20.04	159
Penta CT	26.44	229

E-9 STANDARD PREPARATION

E-9.1 Stock Solution

Weigh 0.002 5 g each standard reagent (**E-7.1** to **E-7.26**) in each 25 ml volumetric flask and fill to the mark with methanol.

Stock solution of each standard is 100 ppm.

E-9.2 Stock Solution No. 1 except 2,6-DCT and 3,4-DCT

Take 1 ml of each stock solution in 100 ml volumetric flask and fill it up to the mark with DCM (1 ppm each mixture solution).

E-9.3 Stock Solution No. 2

Take 10 ml of above stock solution no.1 in 100 ml volumetric flask and fill it up to the mark with DCM (0.1 ppm each mixture solution).

E-9.4 Stock Solution No. 3

Take 5 ml of above stock solution no.1 in 100 ml volumetric flask and fill it up to the mark with DCM (0.05 ppm each mixture solution).

E-9.5 Stock Solution No. 4

Take 1 ml of above stock solution No.1 in 100 ml volumetric flask and fill it up to the mark with DCM (0.01 ppm each mixture solution).

E-9.6 Stock Solution No. 5

Take 10 ml of above stock solution No.3 in 100 ml volumetric flask and fill it up to the mark with DCM (0.01 ppm each mixture solution).

E-10 SAMPLE PREPARATION

Weigh 0.1 g sample into a 10 ml volumetric flask. Add 5 ml DCM and shake well on cyclo mixer. After 30 min fill to the mark with DCM. The solution has to be filtered before injection into the GC-MS.

If necessary dilute the sample solution according to the calibration range.

E-11 CALCULATION

$$\text{CB's, ppm} = \frac{A_2 \text{ x } V_1 \text{ x } W_1 \text{ x } B_2}{A_1 \text{ x } V_2 \text{ x } W_2 \text{ x } B_1} \text{ x } 10^6$$

where

A_1 = area of standard chlorobenzene’s (CB’s);

V_1 = injection volume of standard chlorobenzene’s (CB’s);

W_1 = weight of standard chlorobenzene’s (CB’s);

B_1 = total volume of standard chlorobenzene’s (CB’s);

A_2 = area of chlorobenzene’s (CB’s) peak in sample;

V_2 = injection volume of sample;

W_2 = weight of sample; and

B_2 = total volume of sample.

Similarly, contents of chlorotoluene’s (CT’s) shall be calculated.

E-12 REPORT

Result shall be reported as sum of chlorobenzene’s (CB’s) and chlorotoluene’s (CT’s).

ANNEX F

(*Clause* 5.1)

SAMPLING OF 1-NAPHTHYLAMINE

F-1 GENERAL REQUIREMENTS

F-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

F-1.2 Samples shall be taken at a place protected from damp air, dust and soot.

F-1.3 Sampling instrument shall be clean and dry.

F-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument, and the containers for samples from adventitious contamination.

F-1.5 To draw a representative sample, the contents of each container, selected for sampling, shall be mixed, as thoroughly as possible, by suitable means.

F-1.6 The samples shall be placed in clean, dry and air-tight glass or other suitable containers on which the material has no action.

F-1.7 The sample containers shall be of such a size that they are almost completely filled by the sample.

F-1.8 Each sample containers shall be sealed air-tight after filling and marked with full details of sampling, the date of sampling, year of manufacture, and other important particulars of the consignment.

F-1.9 Samples shall be stored in a cool and dry place.

F-2 SAMPLING INSTRUMENT

F-2.1 The sampling instrument is a closed type sampling tube, undivided, consisting of two concentric cylindrical tubes made of a metal which is not affected by the material being sampled (preferably of stainless steel) closely fitting into each other throughout their entire length so that one tube can be rotated within the other, a suitable handle being provided for the purpose. Longitudinal openings of about one-third the circumference are cut in both tubes throughout their length. In one position the two openings coincide and admit the material into the hollow inner tube. By rotating the inner tube through 180° the opening is tightly closed and a core of material being enclosed therein may be withdrawn. This type of sampler is usually provided with locking arrangement so that the tubes are held together in any desired position. The outer tube is provided with a sharp conical end to facilitate penetration, but the base of the cone shall be closed so that no material is entrapped in this portion. The height of the cone shall be equal to its base diameter. The whole instrument shall be of sufficient length to penetrate an entire diagonal of the container being sampled. The diameter of the inner cylindrical space may vary from 20 to 40 mm proportionately to the length. A length of 150 cm and a diameter of 30 mm can cater for most needs (*see* Fig. 2).

F-2.1.1 *Use of Sampling Instrument*

The instrument is inserted in closed position in an oblique direction till it touches bottom. The material is admitted by rotating and opening the tubes and finally closing them, withdrawing the sample in this process. In case the minimum quantity of material required for test from each container is more than the capacity of the instrument, further 'Cores' shall be taken from different parts of the same container such that they are at least

75 mm in the case of kegs, drums, bags, etc. and 25 mm in the case of small containers, from the wall of the container. In all cases the instrument shall be inserted till it touches bottom so that an entire cross-section is withdrawn.

NOTE — The above type of instrument is suitable for materials which are in the form of powder, flakes and paste. If the material is in the form of lumps, the contents of the drum may be melted by heating it in a steam heated chambers and then withdrawing a sample of the melted product as already indicated above.

F-3 SCALE OF SAMPLING

F-3.1 Lot

All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of containers pertaining to different batches of manufacture, then the containers belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a separate lot.

F-3.2 For ascertaining the conformity of the lot to the requirements of this specification, tests shall be carried out for each lot separately. The number (*n*) of containers to be selected for drawing the samples shall depend upon the size (*N*) of the lot and shall be in accordance with Table 2.

F-3.3 These containers shall be selected at random from the lot and to ensure the randomness of selection, random number tables shall be used. In case, such tables are not available, the following procedure may be adopted:

Starting from any container, count them in one order as 1,2,3...., upto *r* and so on, where *r* is an integral part of *N/n* (*N* being the lot size and *n* the number of containers to be selected). Every r^{th} container thus counted shall be withdrawn to give sample for test.

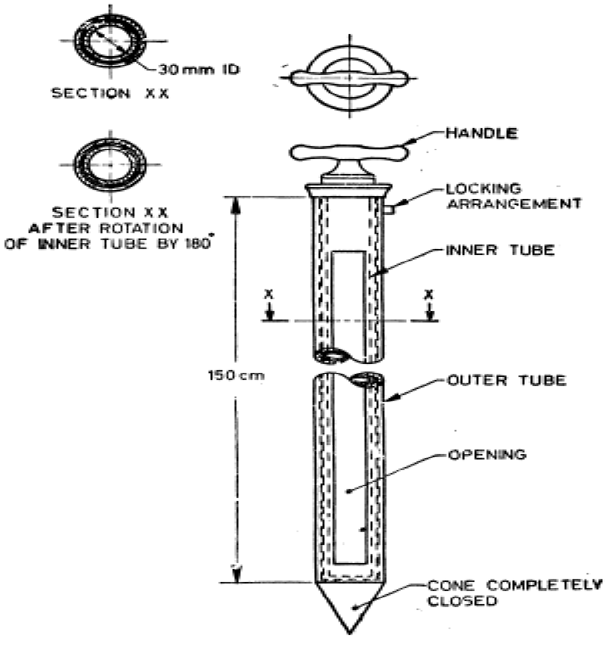


FIG. 2 CLOSED TYPE SAMPLING TUBE, UNDIVIDED

Table 2 Number of Containers to be Selected for Sampling

(*Clause* F-3.2)

Sl No.	Lot Size *N*	No. of Containers to be Selected *n*
(1)	(2)	(3)
i)	4 to 15	3
ii)	16 to 40	4
iii)	41 to 65	5
iv)	66 to 110	6
v)	111 to 200	7
vi)	201 and above	8

NOTE — When the size of the lot is three or less, the number of containers to be selected and the criteria for judging the conformity of the lot to the specification shall be as agreed to between the purchaser and the supplier.

F-4 TEST SAMPLE AND REFERE SAMPLE

F-4.1 From each of the containers selected as in **F-3.2** draw with an appropriate sampling implement small portions of the material from different parts of the container. The total quantity so drawn from each of the containers shall be approximately equal to thrice the quantity required for testing purposes as indicated in **F-4.2**.

F-4.2 Mix thoroughly all the portions of the material drawn from the same container to give a representative sample for the container.

F-4.3 From the samples (*see* **F-4.2**) representing different containers selected in **F-3.2**, a small but equal quantity of material shall be taken and thoroughly mixed to form a composite sample, sufficient to carry out testing for the characteristics specified under **3.2**. The composite sample so obtained shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

F-4.4 The remaining portion of the material in the samples (*see* **F-4.2**) from different containers shall be divided into three equal parts, each forming an individual sample. One set of individual samples representing the *n* containers selected shall be for the purchaser, another for the supplier and the third for the referee.

F-4.5 All the individual and composite samples shall be transferred to separate containers. These containers shall then be sealed air-tight with stoppers and labelled with full identification particulars given in **F-1.8**.

F-4.6 The referee samples consisting of a composite sample and a set of *n* individual samples shall bear the seals of both the purchaser and the supplier and shall be kept at a place agreed to between the two. This shall be used in case of any dispute between the two.

ANNEX G

(*Foreword*)

COMMITTEE COMPOSITION

Dye Intermediates Sectional Committee, PCD 26

Organization	*Representative(s)*
Institute of Chemical Technology, Mumbai	Prof Ganapati Subray Shankarling **(*Chairperson*)**
Aarti Industries Limited, Mumbai	Dr Vaishali Bhandary Dr Sanjeev Kumar Dixit (*Alternate)*
Alkyl Amines Chemicals Limited, Mumbai	Shri S. V. Nikumbhe Shri Kirat Patel *(Alternate)*
Ankleshwar Research and Analytical Infrastructure Limited, Ankleshwar	Shri Mansukh H Vekaria
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Gujarat Pollution Control Board, Gandhinagar, Ahmedabad	Shri Y. A. Tai

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Scientist ‘B’, BIS

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Organization	*Representative(s)*
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Archroma India Private Limited, Thane	DR RAJESH RAMAMURTHY
Central Revenue Control Laboratory, New Delhi	DR T. A.SREENIVASA RAO
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